

MICRO LIQUID-LIQUID EXTRACTION GAS CHROMATOGRAPHIC METHOD SM 20 <sup>th</sup> ED 6640 B					Page 1 of 2
Facility Name: _____					VELAP ID _____
Assessor Name: _____					Analyst Name: _____
					Inspection Date _____
Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Records Examined: SOP Number/ Revision/ Date _____ Analyst: _____					
Sample ID: _____ Date of Sample Preparation: _____ Date of Analysis: _____					
Were samples allowed to equilibrate to room temperature prior to processing?	6640.B.4.a 6251.B.5.a				
Was surrogate added to all samples prior to extraction?	6640.B.6.e				
After the addition of Sodium Sulfate, was enough 20% NaOH to produce pH $\geq$ 12?	6640.B.4.b				
Were samples next allowed to sit for 1 hour at room temperature?	6640.B.4.b				
Was MTBE next added and samples shaken vigorously for 1 minute?	6640.B.4.b				
Were these MTBE layers then discarded?	6640.B.4.b				
Were concentrated Sulfuric Acid, Copper Sulfate, and Sodium Sulfate then added to samples?	6640.B.4.b				
Were samples shaken until salt dissolved?	6640.B.4.b				
Was MTBE next added to samples followed by shaking on a mechanical shaker?	6640.B.4.b				
If diazomethane was used in derivitization, was it used only if it was a strong yellow color?	6640.B.4.c				
Was TMSD used for derivitization only if dalapon was not to be determined?	6640.B.4.d				
If TMSD was used in derivitization, was TMSD added to sample extracts followed by heating at 50°C for 1 hour?	6640.B.4.d				
If TMSD was used in derivitization, after cooling after above step, was 2M acetic acid in methanol added to samples to react excess TMSD?	6640.B.4.e				
Notes/ Comments:					

**MICRO LIQUID-LIQUID EXTRACTION GAS CHROMATOGRAPHIC METHOD II**  
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Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
If diazomethane was used in derivitization, was it added to extracts and was a faint yellow color persistent after addition?	6640.B.4.e				
If diazomethane was used in derivitization, was silica gel added autosampler vials to quench excess diazomethane?	6640.B.4.e				
Prior to each analytical run, did the analysis of MTBE blanks demonstrate that there were no solvent interferences?	6640.B.4.f				
Were at least three calibration standards used?	6640.B.4.g				
Were method blanks of reagent water prepared and extracted each time a set of sample is extracted and each time a reagent is changed?	6640.B.6.b				
Did surrogate recoveries not exceed 100±30%?	6640.B.6.e				
If internal standard was used, did internal standard areas of samples not exceed ±30% of the average internal standard areas of the calibration standards?	6640.B.6.f				
When using internal standard calibration, were response factors (RFs) used for quantitation only if RF values were ≤ 20% RSD?	6640.B.6.j				
When using external standard calibration, were average calibration factors used for quantitation only if the calibration factors were less than 20%RSD?	6640.B.6.j				
Were check standards analyzed at least once each shift to be ±20%?	6640.B.6.j				

Notes/ Comments: